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Patent of exclusion

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[Title in German of the object of the invention:]

Tantallegierung für Spinnndüsen

TANTALUM ALLOY FOR SPINNERETS

Patent Claim

Tantalum alloy for spinnerets, which has a grain-refined alloying contribution, characterized in that the alloy consists of 99.7 to 99.97% [W_B*] by weight of tantalum, 0.01 to 0.10 % by weight niobium and 0.02 to 0.10 % by weight of molybdenum, and the content of usual impurities does not exceed 0.10 % by weight.

*[Translator's note: Throughout the German original text, the applicant/inventors have used abbreviation W_p to indicate the content of alloy in %. Such an abbreviation, W_p , does not exist in the German professional literature. Presumably, it is an abbreviation used by professionals in the now defunct GDR. It is assumed that the inventors have tried to indicate % by weight.]

Title of the Invention

Tantalum alloy for Spinnerets

Area of Application of the Invention

The invention pertains to a tantalum alloy, produced in a melt-metallurgical way, which alloy can be used for spinnerets in the synthetic-fibers industry.

Characteristics of the known Prior Art

Spinnerets of tantalum are used on a large scale in the synthetic-fiber industry. Those spinnerets are deep-drawn [swaged] cups, which are first of all made of a melt-metallurgically produced semi-manufactured product [blank], and, subsequently, are provided with a large number of fine boreholes, having a diameter from 60 to 100 μm , by means of piercing or punching. It is known that the capability of tantalum to pierce

or punch is influenced by the purity of the metal, its hardness, depositions on the grain boundaries, and the grain size.

As is generally known, the melting in electron bombardment furnaces exerts influence upon the purity of the metal. However, in doing so, there are generated grain sizes of up to few millimeters. Therefore, trials have been conducted to process tantalum, produced in a melt-metallurgical way, into a sheet metal [metal-sheet], having a thickness of 0.4 to 0.5 mm and characteristic or pronounced fine grain. In accordance with the D.D.R. Economic patent DD-WP 142120, the tantalum ingot [block] t, which has been smelted in an electron-bombardment furnace, was compressed or forged into a cube as a result of being subjected to upset forging, and after a rotation thru 90°, it is pressed or cold-shaped into a sheet, which subsequently is rolled into a metal-sheet as a result of plastic cold working with the help of interposed high-vacuum annealing. Grain sizes, having an average grain diameter of 0.03 to 0.04 mm, are achieved with the help of that method.

When spinnerets, manufactured of that tantalum metal sheet, are pierced, the hard-metal [cemented metal-carbide] piercing needles, used for this purpose, exhibit a short service life. On the average, 2,000 to 4,000 [die-spinning] nozzles holes can be pierced with the help of a needle. Another possibility for the reduction of the grain size consists in the addition of alloying metals [alloying additives]. The recrystallization properties of

substitutional alloys, based on tantalum, has been investigated by GYPEN and DERUYTTERE (see Journal "Metallkunde" 72, (1981) 8, pp 530-533. Among other things, the influence of molybdenum and niobium upon the anomalous crystal growth, which easily occurs in pure tantalum, melted in an electron-bombardment manner, is studied. As a result of the alloying of molybdenum in ratios between 0.32 to 0.70 % by weight, grain sizes, having an average diameter of 0.06 to 0.09 mm are achieved, when the annealing is carried out at the technologically conventional temperature range of 1,000 to 1,200 °C. With niobium, in its capacity as alloying element, in ratios of 2.0 and 4.2 % by weight, there is formed an average grain diameter of 0.06 mm. Also, with grain sizes, which can be adjusted within the cited concentrations, as a result of the alloying of molybdenum or niobium, an extension of the service-life properties of the piercing needles, used for piercing of the spinnerets, does not take place.

Aim of the Invention

The aim of the invention consists in providing a tantalum alloy, having an adequate capability to pierce, with the help of which tantalum alloy the service life of the hard-metal piercing needles, used for the piercing of spinnerets, is extended.

Explanation of the Substance of the Invention

The objective to provide a tantalum alloy, which has a particularly fine-grained texture, and, as a result of this, is particularly suitable for the process of piercing with the help of hard-metal piercing needles, forms the basis of the invention.

In accordance with the invention the set objective is achieved as a result of the fact that the alloy consists of 99.7 to 99.97% by weight of tantalum and 0.01 to 0.10 % by weight of niobium, and 0.02 to 0.10 % by weight of molybdenum, and the content of conventional impurities does not exceed 0.1 % by weight. When this alloy is processed into a metal sheet, having a thickness of 0.23 to 0.50 mm, in accordance with the known methods for the manufacturing of tantalum sheet-metal, the alloy [composition metal] in accordance with the invention exhibits a particular recrystallization texture in conjunction with an uniform fine-grained texture. An average grain diameter of 0.008 to 0.20 mm is achieved. The particular or special recrystallization texture is characterized by an elevated percentage of crystallites, whose surfaces are arranged so that the crystallographic orientation is parallel to the rolling plane.

When the alloy in accordance with the invention is used for spinnerets, significantly longer periods of service life of the hard-metal piercing needles are attained - namely in the order of 6,000 to 8,000 spinnerets' holes/needle - in comparison to the periods of service life of the piercing needles made of pure

tantalum, used in piercing the holes of the spinnerets. This result cannot be explained with the known regularities of the plastic deformation of metals, having a cubic body-centered [body-centered cubic] lattice. In the metals, having a body-centered cubic [bcc] lattice (e.g., α -Fe, Mo, Nb, Ta, W), the plastic deformation takes preferably place in the {111}- glide (slip) plane in the $\langle 111 \rangle$ -directions (see the textbooks by SCHULZE, G.E.R., Metallphysik [Physics of Metals], Akademieverlag, Berlin, 1974, p 221 and SCHATZ, W., Einführung in die Werkstoffwissenschaft [Introduction in the Materials Science], VEB Deutscher Verlag für die Grundstoffindustrie, Leipzig, 1984, p 337).

A preferred plastic deformation perpendicular to {100}-surfaces is not to be expected. The advantageous effect of the additives of niobium and molybdenum in the cited concentrations consists in the change of the texture, the significant reduction of the crystallite size, and in a modified composition on the crystallite boundaries without a perceptible increase of the hardness and reduction of the ductility (elongation at rupture). As a result of this, the capability of the material to pierce is improved as a result of a minor cold-welding tendency between the tantalum alloy and the hard-metal piercing needle. Moreover, as a consequence of the small crystallite size, the danger of an occurrence of deleterious bending stress for the tip of the piercing needle is reduced. All this leads to an elongation of

the service life of the piercing needles.

Exemplified Embodiment

Using tantalum secondary material and niobium wire, a tantalum alloy, having a content of 0.04 % by weight of niobium and 0.02 % by weight of molybdenum, is melted in an electron bombardment furnace. After a surface treatment, the cast ingot was cold shaped [subjected to plastic cold working] by means of desurfacing. This cold shaping took place on a free-forging [hammer-forging press] whereby the cast ingot was forged [subjected to upset forging], and after a rotation thru 90° was pressed into a sheet [plate]. During the mechanical working [or shaping, the ingot [block] was kept at a room temperature by being cooled. After an additional surface treatment, the sheet was rolled into a strip, having a thickness of 3 mm. After a total cold face [cold] working of 98.5%, a recrystallization in high vacuum took place at a temperature of 1,100 °C/1 hr. After the rolling on the end thickness, there took place a final high-vacuum annealing at 1,200 °C/1 hr or 1,000 °C/2h. The results of the roentgenographical (X-ray photographic) texture analysis speak of an elevated percentage of the texture component (100) [110] in comparison to pure tantalum.

An average grain diameter of 0.015 mm was attained. The spinneret blanks, made of this alloy, are characterized by a

particularly adequate processing properties. With the help of a hard-metal piercing needle, the manufacturing of 6,240 [spinneret-]nozzle holes in a spinneret was possible.

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